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L3: Entry 1 of 3

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Nov 24, 1998

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DOCUMENT-IDENTIFIER: JP 10310508 A

TITLE: MODIFIED POWDER, AND COSMETIC CONTAINING THE SAME

PUBN-DATE: November 24, 1998

## INVENTOR-INFORMATION:

NAME

COUNTRY

TACHIBANA, KIYOMI

INT-CL (IPC): A61 K 7/02

## ABSTRACT:

PROBLEM TO BE SOLVED: To obtain a treated powder capable of providing a cosmetic excellent in sebum resistance, water resistance and durability by formulating with a cosmetic by carrying out a surface treatment with a specific silicone compound modified with a fluorinated alkyl in combination with a (poly)glycerin.

SOLUTION: This treated powder is obtained by carrying out a surface treatment of (A) an inorganic or organic powder optionally subjected to a silicone treatment, a pendant treatment, etc., with (B) 0.1-100 wt.% silicone compound modified with a fluorinated alkyl in combination with a (poly)glycerin, of formula I {R1 to R12 is each a 1-10C alkyl or a phenyl with the proviso that one or more of the R1 to R12 is a fluorine-substituted alkyl, and further another one or more thereof is formulas II to IV [Q is a 1-10C divalent hydrocarbon; (1) is 1-20; (m) is 1-20]; (p), (q) and (r) each  $\geq 0$ . and when (p)=(q)=0 and (r) $\neq 0$ , one or more of R1 to R3 and R8 to R12 are each a fluorine-substituted alkyl and formulas II to IV, and when (p)=(r)=0 and (q) $\neq 0$ , one or more of R1 to R3, R6 to R7 and R10 to R12 are each a fluorine-substituted alkyl and formula II to IV} based on the component A.

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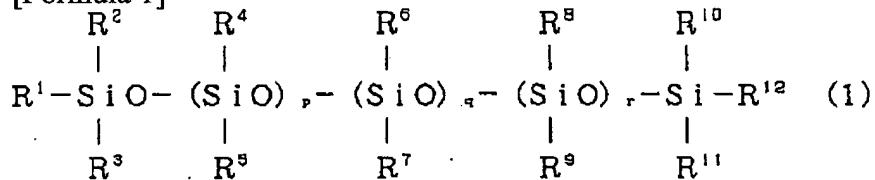
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## CLAIMS

## [Claim(s)]

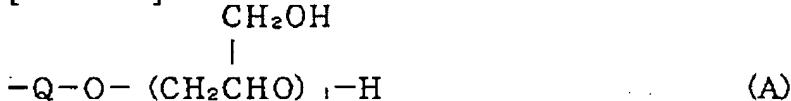
[Claim 1] Refining fine particles characterized by carrying out surface treatment with the fluorine alkyl glycerol (Pori) covariance silicone compound shown by the following general formula (1).

## [Formula 1]

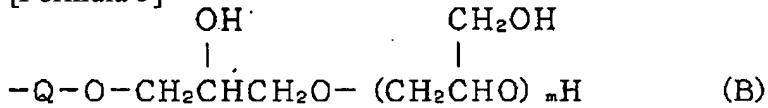


the inside of [type, and R1-R12 are the same -- or -- you may differ -- the alkyl group of carbon numbers 1-10, and a phenyl group -- being shown -- at least one [ among those, ] -- a fluorine permutation alkyl group -- it is -- at least one [ furthermore, ] -- the following formula (A) and (B) -- or (C) --

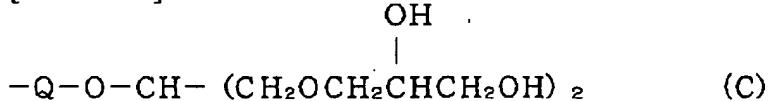
## [Formula 2]



## [Formula 3]



## [Formula 4]



(-- however, Q is [ 1-20m of the divalent hydrocarbon group of carbon numbers 1-10 and 1 ] the positive integers of 1-20.), although the radical shown, and p, q and r are zero or more integers, respectively At least one of R1-R3, and the R8-R12 at the time of p=q=0 and r!=0 A fluorine permutation alkyl group, The radical expressed with the above-mentioned formula (A), (B), or (C) is shown. At least one of R1-R3, R6-R7, and the R10-R12 at the time of p=r=0 and q!=0 And a fluorine permutation alkyl group, The radical expressed with the above-mentioned formula (A), (B), or (C) is shown. At least one of R1-R5, and the R10-R12 at the time of p!=0 and q=r=0 And a fluorine permutation alkyl group, The radical expressed with the above-mentioned formula (A), (B), or (C) is shown. At least one of R1-R3, and the R6-R15 at the time of p= 0, q=r!=0, and \*\* And a fluorine permutation alkyl group, The radical expressed with the above-mentioned formula (A), (B), or (C) is shown. At least one of R1-R5, and the

R8-R12 at the time of  $p=r=0$  and  $q=0$  And a fluorine permutation alkyl group, The radical expressed with the above-mentioned formula (A), (B), or (C) is shown. At least one of R1-R7, and the R10-R12 at the time of  $p=q=0$  and  $r=0$  And a fluorine permutation alkyl group, and the radical expressed with the above-mentioned formula (A), (B), or (C) is shown, and at least one of R1-R3, and the R10-R12 shows the radical expressed with a fluorine permutation alkyl group and the above-mentioned formula (A), (B), or (C) at the time of  $p=q=r=0$ . ]

[Claim 2] The charge of makeup characterized by containing refining fine particles according to claim 1.

[Translation done.]

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**DETAILED DESCRIPTION**

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**[Detailed Description of the Invention]****[0001]**

**[Field of the Invention]** This invention relates to the charge of makeup excellent in water-repellent oil repellency and endurance which blended it with the refining fine-particles list by which surface treatment was carried out with the fluorine alkyl glycerol (Pori) covariance silicone compound which has simultaneously the fluorine compound content radical which has water repellence, and a hydrophilic radical.

**[0002]** In more detail, when a fluorine alkyl glycerol (Pori) covariance silicone coated powder object holds water, without losing water-repellent oil repellency, the fine particles itself are using the phenomenon of coming to have adhesion with the skin, and it is related with the charge of makeup excellent in sebaceous one-proof, a water resisting property, and endurance.

**[0003]**

**[Description of the Prior Art]** As it was in JP,61-55481,B conventionally, by carrying out surface treatment of the fine particles with various fluorine compounds, the fluoro alkyl chain was introduced into the fine-particles front face, the property of water-repellent oil repellency was added to fine particles, and development of the charge of makeup which was excellent at makeup durability using fine particles getting wet neither in sebum nor sweat was performed.

**[0004]**

**[Problem(s) to be Solved by the Invention]** Here, although physical adhesion force took the lead in many cases, association with fine particles and the skin could not maintain those association to the sweat of a large quantity, and sebum, but passed as a result, and the problem that the makeup film became thin by the time generated it.

**[0005]** On the other hand, as it was in JP,6-102607,B, combining fluoridization fine particles and fluorine system oils was also considered, but bonding strength with the skin was raised by leaps and bounds, and by the time it developed the charge of makeup excellent in makeup durability, it did not result.

**[0006]**

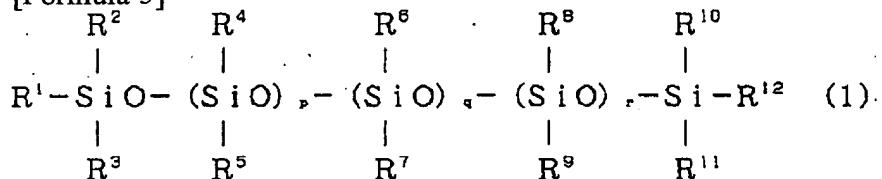
**[Means for Solving the Problem]** As a result of inquiring wholeheartedly in view of the above-mentioned actual condition, it has the property in which the fluorine alkyl glycerol (Pori) covariance silicone compound held water and which is case [ a property ], thickened or gelled, It is giving having strong water-repellent oil repellency, after holding the water of a constant rate to a header, and giving this property to fine particles by surface treatment. Although it finds out that the charge of makeup excellent in sebaceous one-proof, a water resisting property, and endurance is obtained, a fluorine alkyl glycerol (Pori) covariance silicone compound works as some oils on the front face of fine particles in the case of the charge of makeup which does not use water further and a role of a feel modifier at the time of spreading is played After moisture was supplied from fixed time amount and the skin, the knowledge of playing the role for the improvement in makeup durability was acquired by thickening and increasing bonding strength with the skin.

[0007] A fluorine alkyl glycerol (Pori) covariance silicone compound Moreover, a fluorine alkyl part, The surface treatment which has such constituents and compatibility since it has a glycerol part and a silicone part, (Pori) Use the surface hydroxyl group of unsettled fine particles, or For example, fluorine compound processing, siliconization, After carrying out simultaneously with amino acid processing etc. or performing those surface treatment in advance, also by carrying out surface treatment of the fluorine alkyl glycerol (Pori) covariance silicone compound It finds out that a feel side and the description same in respect of durability are acquired, and came to complete this invention.

[0008] That is, this inventions are the refining fine particles by which surface treatment was carried out with the fluorine alkyl glycerol (Pori) covariance silicone compound shown by the following general formula (1), and a charge of makeup containing it.

[0009]

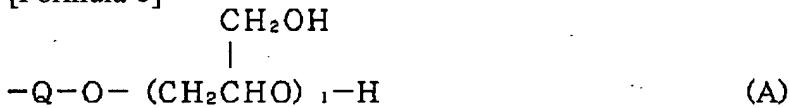
[Formula 5]



[0010] the inside of [type, and R1-R12 are the same -- or -- you may differ -- the alkyl group of carbon numbers 1-10, and a phenyl group -- being shown -- at least one [ among those, ] -- a fluorine permutation alkyl group -- it is -- at least one [ furthermore, ] -- the following formula (A) and (B) -- or (C) --

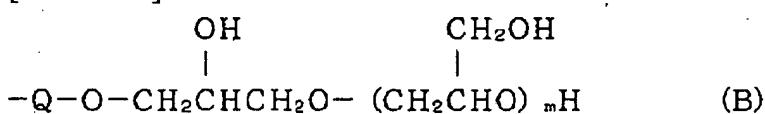
[0011]

[Formula 6]



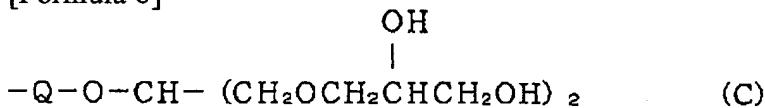
[0012]

[Formula 7]



[0013]

[Formula 8]



[0014] ( -- however, Q is [ 1-20m of the divalent hydrocarbon group of carbon numbers 1-10 and 1 ] the positive integers of 1-20.), although the radical shown, and p, q and r are zero or more integers, respectively At least one of R1-R3, and the R8-R12 at the time of p=q=0 and r!=0 A fluorine permutation alkyl group, The radical expressed with the above-mentioned formula (A), (B), or (C) is shown. At least one of R1-R3, R6-R7, and the R10-R12 at the time of p=r=0 and q!=0 And a fluorine permutation alkyl group, The radical expressed with the above-mentioned formula (A), (B), or (C) is shown. At least one of R1-R5, and the R10-R12 at the time of p!=0 and q=r=0 And a fluorine permutation alkyl group, The radical expressed with the above-mentioned formula (A), (B), or (C) is shown. At least one of R1-R3, and the R6-R15 at the time of p= 0, q=r!=0, and \*\* And a fluorine permutation alkyl group, The radical expressed with the above-mentioned formula (A), (B), or (C) is shown. At least one of R1-R5, and the R8-R12 at the time of p=r!=0 and q= 0 And a fluorine

permutation alkyl group, The radical expressed with the above-mentioned formula (A), (B), or (C) is shown. At least one of R1-R7, and the R10-R12 at the time of  $p=q=0$  and  $r=0$  And a fluorine permutation alkyl group, and the radical expressed with the above-mentioned formula (A), (B), or (C) is shown, and at least one of R1-R3, and the R10-R12 shows the radical expressed with a fluorine permutation alkyl group and the above-mentioned formula (A), (B), or (C) at the time of  $p=q=r=0$ . ] Hereafter, this invention is explained to a detail.

[0015] the fluorine alkyl glycerol (Pori) covariance silicone compound used by this invention is shown in the above-mentioned general formula (1) -- as -- a mono-glycerol -- and -- or it is the compound which polyglycerin combined with the silicone chain through the spacer. Or you may differ. in the compound shown by the above-mentioned general formula (1), R1-R12 are the same -- The alkyl group of carbon numbers 1-10 and a phenyl group are shown. For example, a methyl group, An ethyl group, a propyl group, butyl, a pentyl radical, a hexyl group, a heptyl radical, An octyl radical, a nonyl radical, a DEKERU radical, a cyclopentylic group, a cyclohexyl radical, A phenyl group, a tolyl group, etc. are mentioned and at least one of R1-the R12 is a fluorine permutation alkyl group. For example, although it is the radical with which a trifluoro propyl group, a nona fluoro hexyl group, a heptadeca fluoro decyl group, etc. are mentioned, and also at least one of R1-the R12 is indicated to be by the above-mentioned formula (A), (B), or (C) A mono-glycerol, diglycerol, triglycerol, a tetra-glycerol, etc. are mentioned.

[0016] As fine particles by which surface treatment is carried out with the fluorine alkyl glycerol (Pori) covariance silicone compound besides shown in an account general formula (1), inorganic [ which be conventionally used for cosmetics ] or organic fine particles is pointed out, and macromolecules, such as lake coloring matter, a color, nylon powder, and silk powder, a colored pigment, white pigments, an extender, a pearl pigment, a metal salt, inorganic fine particles, metallic soap, a particle zinc oxide, particle titanium oxide, particle ferrous oxide, alumina processing particle titanium oxide, silica processing particle titanium oxide etc. mentioned Simultaneously with the above-mentioned general formula (1), after [ general ] carrying out surface treatment and carrying out general surface treatment, surface treatment of these fine particles may be further carried out by the above-mentioned general formula (1).

[0017] As a class of surface treatment, well-known surface treatment, for example, fluorine compound processing, siliconization, pendant processing, silane coupling agent processing, titanium coupling agent processing, oils processing, N-acylation lysine processing, polycarboxylic acid processing, metallic soap processing, amino acid processing, inorganic compound processing, living body extract component processing, plasma treatment, mechanochemical processing, etc. are mentioned conventionally.

[0018] As the approach of surface treatment, a wet method, dry process, the mechanochemical method, etc. are mentioned.

[0019] Although the mixed rate with the fluorine alkyl glycerol (Pori) covariance silicone compound, unsettled fine particles, or processing fine particles in surface treatment changes with the oil absorption and specific surface area of fine particles, 0.1 - 100 % of the weight (it is only hereafter described as "%") is 1 - 15% desirable still more preferably to the weight of unsettled fine particles or processing fine particles. If less than 0.1% of processing is sometimes insufficient and it exceeds 100%, a feel may worsen.

[0020] Although it changes with pharmaceutical forms of the charge of makeup when blending the refining fine particles of this invention with the charge of makeup, it is desirable to blend 0.1 - 99% for refining fine particles to the charge of makeup.

[0021] All the products applied externally as a charge of makeup of this invention by the skin by which a feel is made a problem at the time of the activity of charges of skin makeup, such as charges of makeup makeup, such as foundation, face powder, presto powder, a teak color, a substrate, a lip stick, eye shadow, an eyeliner, and a nail color, a cream, a milky lotion, and face toilet, and not only the charge of hair makeup but a powder spray, external use drugs, etc. are included.

[0022] In addition to the above-mentioned component, at the charge of makeup of this invention, the solid-state used for the charge of makeup usual in the range which does not bar the effectiveness of this invention, a semisolid, liquefied oils, water, a water soluble polymer, polyhydric alcohol, a solvent, a

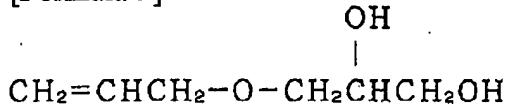
surfactant, fine particles, resin, an organic denaturation clay mineral, a macromolecule, an ultraviolet ray absorbent, a moisturizer, antiseptics, a germicide, perfume, an antioxidant, the component for lustrous skin, a bioactive component, etc. can be blended.

[0023]

[Example] This invention is not limited by these examples, although an example is given to below and this invention is explained to it.

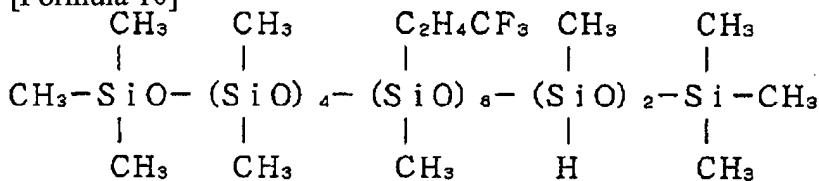
[0024] It is glycerol monoallyl ether 38.4g of the following type, and [0025] to the glass flask which attached example of manufacture 1 agitator, the thermometer, and the reflux condenser.

[Formula 9]



[0026] And SiH radical content fluorine alkyl denaturation silicone 200g expressed with the following type, [0027]

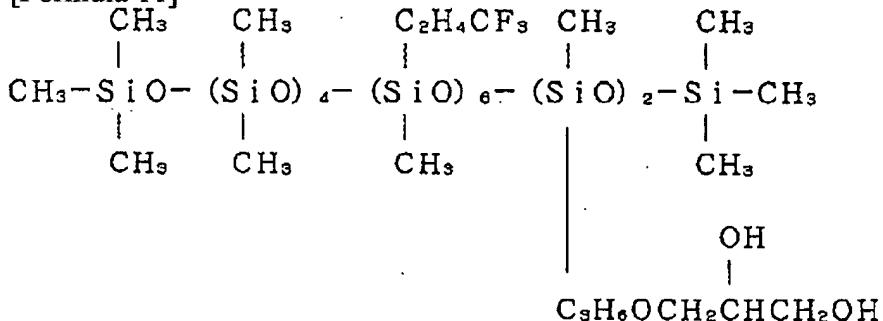
[Formula 10]



[0028] Isopropyl alcohol 100g, 0.3g of 10% ethanol solutions of potassium acetate, and 0.3g (2% of platinum concentration) of isopropyl alcohol solutions of chloroplatinic acid were prepared and heated, and the reaction was performed at the reflux temperature of isopropyl alcohol for 5 hours. Reduced pressure distilling off of the isopropyl alcohol was carried out after reaction termination, and silicone 211.4g of the fluorine alkyl glycerol covariance of the following formula was obtained.

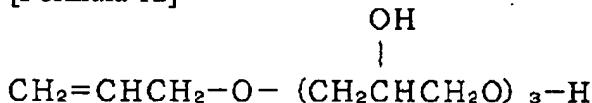
[0029]

[Formula 11]



[0030] It is triglycerol monoallyl ether 81.4g [0031] of the following type about the glycerol monoallyl ether of the example 1 of example of manufacture 2 manufacture.

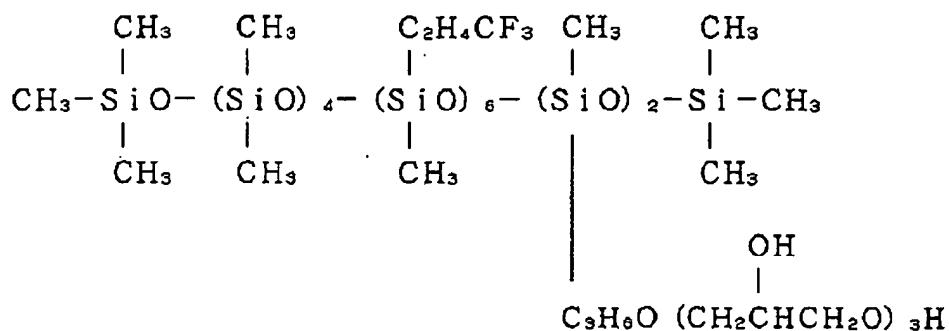
[Formula 12]



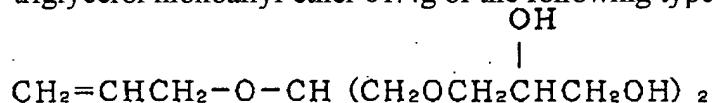
[0032] Except it having been alike and having replaced with, same actuation was performed and silicone 246.6g of the fluorine alkyl polyglycerin covariance of the following type was obtained.

[0033]

[Formula 13]



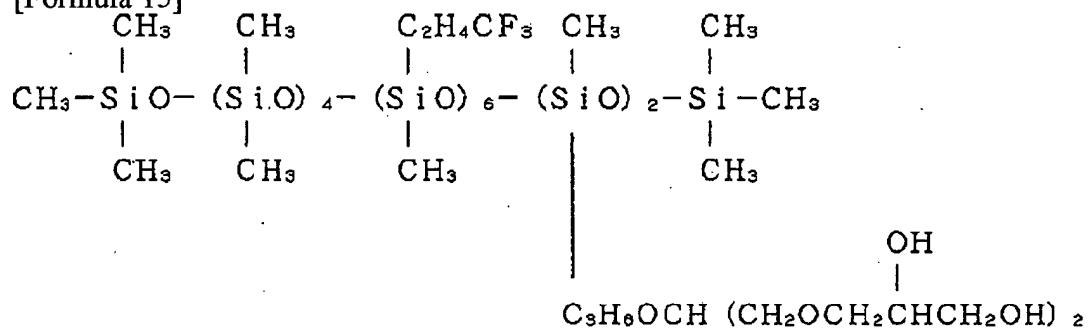
[0034] the glycerol monoallyl ether of the example 1 of example of manufacture 3 manufacture -- triglycerol monoallyl ether 81.4g of the following type -- [Formula 14]



[0035] Except it having been alike and having replaced with, same actuation was performed and silicone 246.6g of the fluorine alkyl polyglycerin covariance of the following type was obtained.

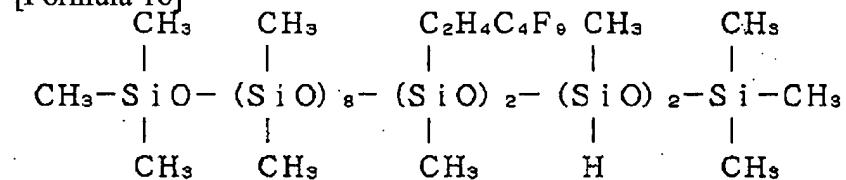
[0036]

[Formula 15]



[0037] SiH radical content fluorine alkyl denaturation silicone 196.3g expressed with the following formula in the SiH radical content fluorine alkyl denaturation silicone of the example 1 of example of manufacture 4 manufacture [0038]

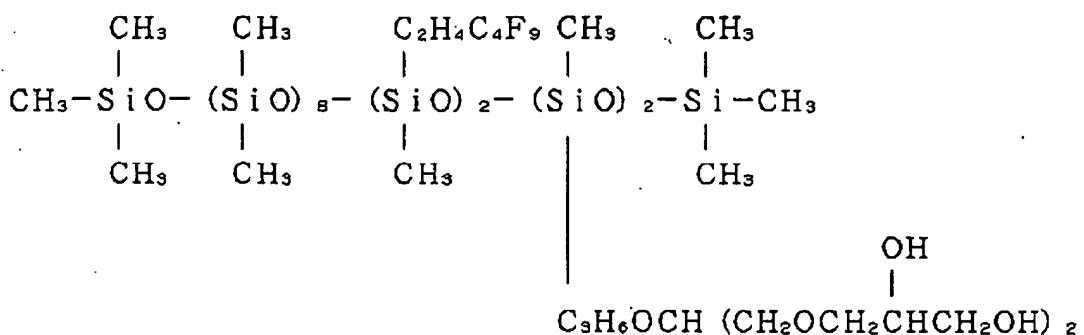
[Formula 16]



[0039] Except it having been alike and having replaced with, the same actuation as the example 3 of manufacture was performed, and silicone 243g of the fluorine alkyl polyglycerin covariance of the following type was obtained.

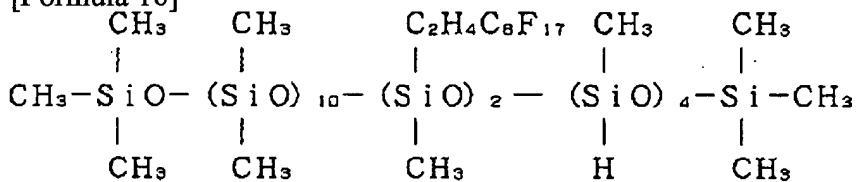
[0040]

[Formula 17]



[0041] SiH radical content fluorine alkyl denaturation silicone 142.3g expressed with the following formula in the SiH radical content fluorine alkyl denaturation silicone of the example 1 of example of manufacture 5 manufacture [0042]

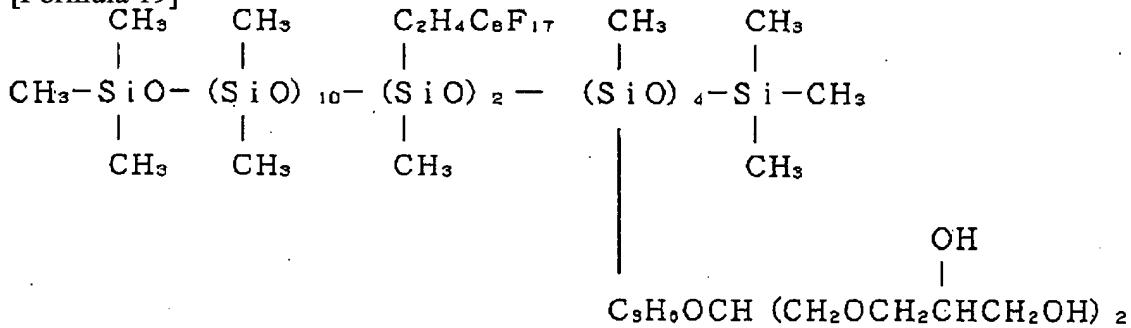
[Formula 18]



[0043] Except it having been alike and having replaced with, the same actuation as the example 3 of manufacture was performed, and silicone 194.7g of the fluorine alkyl polyglycerin covariance of the following type was obtained.

[0044]

[Formula 19]



[0045] After making the chloroform 300 section distribute the example 1 sericite 95 section, the fluorine alkyl glycerol denaturation silicone compound 5 section obtained in the example 3 of manufacture is added and stirred. It ground and the refining sericite was obtained, after carrying out reduced pressure distilling off of the chloroform. Similarly, 5% fluorine alkyl glycerol covariance silicone compound processing titanium oxide, talc, yellow oxide of iron, red ochre, and black oxide of iron were obtained

[0046] After making the chloroform 300 section distribute the 23% methyl-hydrogen-polysiloxane processing sericite 92 of examples section, the fluorine alkyl polyglycerin covariance silicone compound 8 section obtained in the example 2 of manufacture is added and stirred. It ground and the refining sericite was obtained, after carrying out reduced pressure distilling off of the chloroform. Similarly, 8% fluorine alkyl polyglycerin covariance silicone compound and siliconization titanium oxide, talc, yellow oxide of iron, red ocher, and black oxide of iron were obtained.

[0047] After making the chloroform 300 section distribute the 5% processing sericite 96 of example 3 perfluoro alkyl phosphate section, the fluorine alkyl polyglycerin covariance silicone compound 4 section obtained in the example 1 of manufacture is added and stirred. It ground and the refining sericite was obtained, after carrying out reduced pressure distilling off of the chloroform. Similarly, 4% fluorine alkyl polyglycerin covariance silicone compound and perfluoroalkyl processing titanium oxide, talc.

yellow oxide of iron, red ocher, and black oxide of iron were obtained.

[0048] examples 4-6 and example 1 of a comparison foundation -- the foundation of each presentation shown in the following table 1 was manufactured, and it evaluated about the usability.

[0049]

[A table 1]

(%)

(成分)	実施例			比較例
	1	2	3	1
1.処理珪藻(実施例1)	42.0	—	—	—
2.処理珪藻(実施例2)	—	42.0	—	—
3.処理珪藻(実施例3)	—	—	42.0	—
4.未処理珪藻	—	—	—	42.0
5.処理酸化カソ(実施例1)	12.0	—	—	—
6.処理酸化カソ(実施例2)	—	12.0	—	—
7.処理酸化カソ(実施例3)	—	—	12.0	—
8.未処理酸化カソ	—	—	—	12.0
9.処理タル(実施例1)	24.0	—	—	—
10.処理タル(実施例2)	—	24.0	—	—
11.処理タル(実施例3)	—	—	24.0	—
12.未処理タル	—	—	—	24.0
13.処理黄酸化鉄(実施例1)	2.4	—	—	—
14.処理黄酸化鉄(実施例2)	—	2.4	—	—
15.処理黄酸化鉄(実施例3)	—	—	2.4	—
16.未処理黄酸化鉄	—	—	—	2.4
17.処理ベニガラ(実施例1)	0.8	—	—	—
18.処理ベニガラ(実施例2)	—	0.8	—	—
19.処理ベニガラ(実施例3)	—	—	0.8	—
20.未処理ベニガラ	—	—	—	0.8
21.処理黒酸化鉄(実施例1)	0.3	—	—	—
22.処理黒酸化鉄(実施例2)	—	0.3	—	—
23.処理黒酸化鉄(実施例3)	—	—	0.3	—
24.未処理黒酸化鉄	—	—	—	0.3
25.シリカゲル(時計)	2.0	2.0	2.0	2.0
26.流动パラフィン	残量	残量	残量	残量
27.シリコン樹脂、カーボン	8.0	8.0	8.0	8.0
28.香料	適量	適量	適量	適量
29.防腐剤	適量	適量	適量	適量

[0050] (The manufacture approach)

A: Mix components 1-24 by the HENSHIRU mixer.

B: Mix components 25-29 and, in addition to A, stir further.

C: After the atomizer ground, the \*\* type of C was carried out to metal mold, and foundation was obtained.

[0051] (The assessment approach) 50 women's panel performed the activity test, the following criteria estimated the mileage to the skin, adhesive goodness, a lack [ stickiness ], the beauty of a result, and the merit of makeup \*\*\*\*, and it judged in the average mark.

[Valuation basis]

five point: -- dramatically -- four fitness : three fitness: -- usually -- two point: -- a little -- defect 1 point: -- a defect [a judgment]

O more than :average mark 4.5O -- a less than 2.5 :average-mark [ or more 3.5 / less than 4.5 ]

\*\*:average-mark [ or more 2.5 / less than 3.5 ] x:average mark profit \*\*\*\* result is shown in a table 2.

[0052]

[A table 2]

(評価項目)	実施例			比較例
	4	5	6	1
肌へののび	○	○	○	×
肌への付着性	○	○	○	○
べたつきのなさ	○	○	○	×
仕上がりの美しさ	○	○	○	△
化粧持続の良さ	○	○	○	×
総合評価	○	○	○	×

[0053] The examples 1-3 which blended the fine particles processed with the fluorine alkyl glycerol (Pori) covariance silicone compound concerning this invention so that clearly from the result of a table 2 are excellent in the mileage to the skin, and adhesion compared with the example 1 of a comparison which blended unsettled fine particles, and do not have stickiness, and it is the beautiful foundation of a result, and it turned out that makeup \*\*\*\* is also very good.

[0054]

[Effect of the Invention] As explained in full detail above, the charge of makeup which blended the refining fine particles by which surface treatment was carried out with the fluorine alkyl glycerol (Pori) covariance silicone compound is excellent in water-repellent oil repellency and endurance, and is the very good thing of makeup \*\*\*\*.

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DERWENT-ACC-NO: 1999-064788

DERWENT-WEEK: 200413

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TITLE: Cosmetic powder - has surface treated with fluorine alkyl (poly)glycerin-codenatred silicone compound

PRIORITY-DATA: 1997JP-0136116 (May 9, 1997)

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## PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
<input type="checkbox"/> <a href="#">JP 3496133 B2</a>	February 9, 2004		008	A61K007/02
<input type="checkbox"/> <a href="#">JP 10310508 A</a>	November 24, 1998		008	A61K007/02

INT-CL (IPC): [A61 K 7/02](#)

ABSTRACTED-PUB-NO: JP 10310508A

## BASIC-ABSTRACT:

A quality-improving powder whose surface is treated with fluorine alkyl (poly)glycerin-codenatredsilicone compound represented by the formula (1) is claimed. R 1 - R 12=the same or different alkyl and phenyl having 1-10 carbons, at leastof which is represented by the formulae, (A), (B) or (C). Q=divalent hydrocarbon having 1-10 carbons. 1 and m=integral numbers of 1-20. Each p, q and r=integral number larger than 0, at least one of R 1 -R3 and R 8 -R 12 is fluorine-substituted alkyl when p=0, q=0 and r is not 0, at least one ofR 1 -R3 ,R6 -R7 and R 10-R12 is fluorine-substituted alkyl or radical of (A), (B)or (C) when p=r=0 and q is not , at least one of R1 -R5 , and R 10-R12 is fluorine-substituted alkyl or radical of (A), (B) or (C) when p is not 0, and q=r=0, at least one of R1 -R3 andR 6 -R15 is fluorine-substituted alkyl or radical of (A), (B) or (C) when p=0, and q and r are not 0, atleast one of R 1 -R5 and R 8 -R12 is fluorine-substituted alkyl or radical of (A), (B) or (C) when p and r are not 0 and q=0, atleast one of R 1 -R7 and R 10-R12 is fluorine-substituted alkyl or radical of(A), (B) or (C) when p and q are not 0 and r=0, and at least oneof R1 - R 3 and R 10-R12 is fluorine-substituted alkyl or radical of(A), (B) or (C) when p=q=r=0 A cosmetic containing the present quality-improving powder is also claimed.

ADVANTAGE - The present cosmetic is resistant against skin fat and water, and has durability.

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